

60-sec averaging time. An analog set-up utilizing a lock-in and a seven-decade ratio transformer, followed by a 12-bit digitizer, is used to measure pressure with 0.1 Pa resolution and long-time stability. All the measurements are automated using a GPIB interface with optical extender to measuring devices inside the shielded room and application programs are written in Labview.

More Preparation for the $\nu = 5/2$ FQHE Experiment

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In our recent report,¹ the true quantum Hall state at $\nu = 5/2$ was observed at ultra-low temperatures using a specially designed sample cooling system.² In this case the 2DEG samples were restricted to a direction perpendicular to the magnetic field. Detailed studies of the anisotropy must be determined in the future measurements, but to achieve tilting of a sample at ultra-low temperature remains a challenge.

We have modified the sample cooling system as shown in Figure 1. In the newly designed cell, one sample is set on a rotator. The rotator is connected to a flexible bellows and

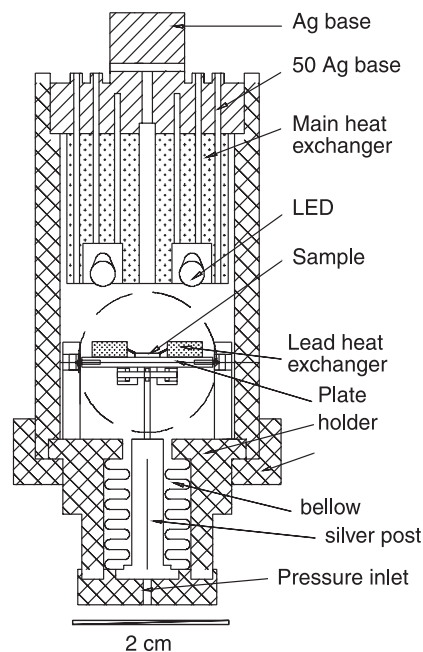


Figure 1. Experimental cell.

turned to the vertical direction from the horizontal employing a pressure of few bars. To minimize the frictional heating the ruby and sapphire bearings are used for the moving parts. The cell and rotator have been tested at liquid N₂ temperatures. This cell will be used in next ultra-low temperature high field measurement of the FQHE effect at large filling factors.

¹ Pan, W., *et al.*, Phys. Rev. Lett., **83**, 3530 (1999).

² Xia, J.S., Proc. LT-22, Helsinki, Finland, August 1999.

ENGINEERING MATERIALS

Fatigue Characterization of C15760 Related to the 100 T Magnets

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The effort on materials development programs related to the 100 T non-destructive magnet has recently concentrated on (a) development of a fabrication route for large cross-section of C15715 (Cu+0.3wt% Al₂O₃) and C15760 (Cu+1.1wt% Al₂O₃) conductors in collaboration with OMG America and IGC Superconductor and (b) investigation of the physical properties and microstructure of above composite. We have emphasized the need to develop various fabrication routes capable of producing the conductors with homogeneous mechanical and

electrical properties and large cross-sections (7.2 mm x 5.2 mm, 10.5 mm x 5.5 mm and 1.3 mm x 9 mm; the corner radius is 1.6 mm). The length of the conductor should be at least 200 m. Two candidate materials to be used for outer coils in the 100 T magnet are C15715 and C15760 conductors in addition to others. The selection of the conductors is based on their mechanical properties and electrical properties at both 295 K and 77 K. The feasibility of the fabrication route and the cost of manufacturing the materials must also be considered. The final fabrication routes for cold deformation were designed by NHMFL and IGC superconductor.

The efforts to produce C15760 wires of the required properties have been successful. In order to achieve the maximum strain hardening no intermediate heat treatment was used. In one of the fabrication routes, the extruded materials were cold drawn from rods of 13.7 mm (0.57 inches) in diameter to rectangular wires of 5.5 mm x 10.5 mm x 1.6 mm corner radius. The drawing strain was 1.3 (reduction-in-area=73%). The materials

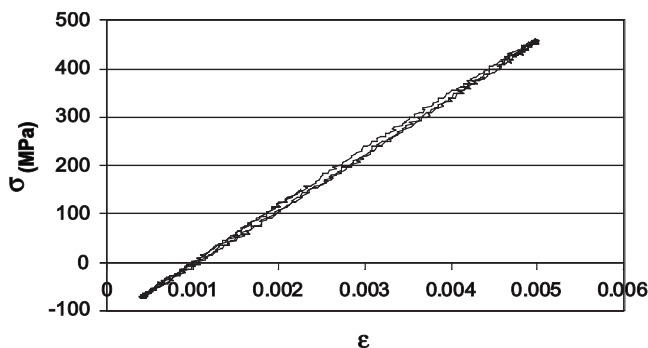


Figure 1. Shape of hysteresis loops of C15769 at 295 K and magnitude of the total strain in fatigue tests. ϵ is the strain and σ is the stress.

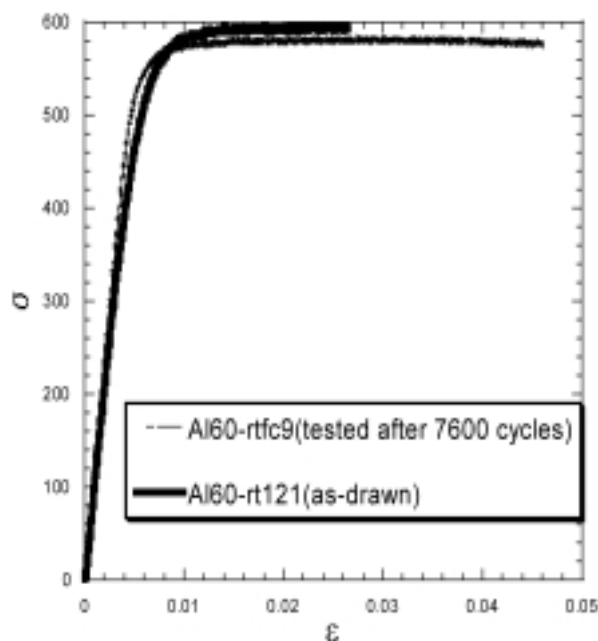


Figure 2. Comparison of the tensile tests from the samples made from C15760 in as-drawn condition and after loaded for about 7600 cycles. Notice that the rounding of the stress-strain curve (which relates to the internal stresses stored in the materials) of the materials after the fatigue is less pronounced than that from as-drawn materials.

can be used for winding coils 1&2 for the outer coils of 100 T magnet.

The study of properties of the conductors has been guided by both design requirements and the service life of magnet. Thus, we have given some consideration both to the role of deformation on the elastic-plastic transition and the mechanical stability of drawn wires made of C15760. The apparent Young's Modulus (E) obtained at 295 K and 77 K are 107 and 126 GPa, respectively. The values of true yield and tensile strength tested at 295 K of the as-drawn C15760 are 553 ± 6 MPa and 587 ± 10 MPa, respectively, whereas the true yield and tensile strengths tested at 77 K are 722 ± 6 MPa and 830 ± 52 MPa (the lowest tensile strength is 800 MPa), respectively. The cyclic softening tests were designed according to the operational mode of the magnet and undertaken in partially reversed tensile-

compression cycles in a strain-controlled mode. The hysteresis loops of C15760 obtained at 295 K are shown in Figure 1, which demonstrates that the tensile true stress is significant higher than compressive stress and material is in a quasi-steady state of deformation. The loop itself, however, also indicates that internal stresses were stored in the materials and instability may occur, as indicated in Figure 2. Under the experimental conditions applied, the shape of the stress-strain curve indicates internal stresses were partially removed after the cyclic tests and the material softens marginally.

Electrical Properties of High Temperature Insulation Coatings by the Sol-Gel Method for Magnet Technology

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The ZrO_2 based coatings have been applied on superconducting tapes and wires using sol-gel technique for high field magnets at NHMFL.¹ Pancake coils are wound from Ag and AgMg/Bi-2212 superconducting tapes in which a "wind and react" (W&R) approach is used due to the brittle nature of the materials. Therefore, it is necessary to use high temperature compatible insulating materials for turn-to-turn electrical insulation in magnets built from the high temperature superconductors (HTS). In this work, the electrical properties of ZrO_2 , MgO-ZrO_2 , $\text{Y}_2\text{O}_3\text{-ZrO}_2$, $\text{CeO}_2\text{-ZrO}_2$, $\text{Sm}_2\text{O}_3\text{-ZrO}_2$, $\text{Er}_2\text{O}_3\text{-ZrO}_2$, $\text{In}_2\text{O}_3\text{-ZrO}_2$, and $\text{SnO}_2\text{-ZrO}_2$ insulation coatings (Figure 1 (a) and (b) that were applied on Ag or AgMg/Bi-2212 tapes, Cu-Nb₃Sn wires, stainless steel and Ni tapes by a continuous, reel-to-reel sol-gel technique were studied. Evaluation of sol-gel insulation using SrTiO_3 , PbTiO_3 , CeO_2 , YSZ, MgO LaAlO_3 and BaZrO_3 base solution were showed no crack, smooth surface as seen in Figure 1(c) and (d).

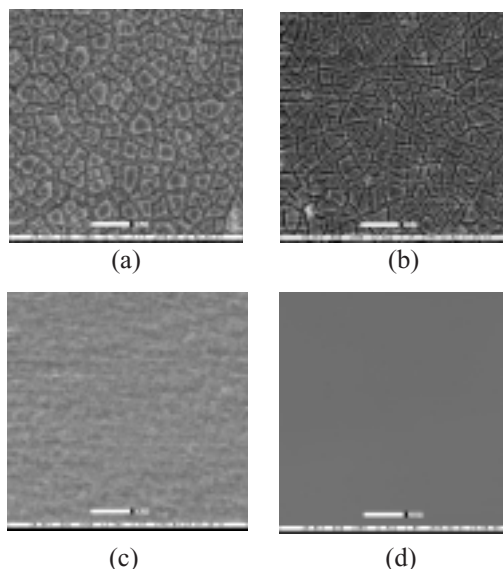


Figure 1. SEM micrographs of the surface of (a) ZrO_2 and (b) MgO-ZrO_2 on AgMg/Bi-2212 tape, (c) BaZrO_3 and (d) YSZ on Ni tapes.

Table 1 shows the capacitance and dielectric constant values of the sol-gel MgO-ZrO₂ insulation coatings measured using a capacitance sandwich with and without epoxy. It was seen that dielectric constant is decreasing as the thickness of the coating goes down. This is probably due to increased porosity and/or higher values of crack space. Table II shows the high voltage breakdown values for the very thin pinhole-free, crack-free insulation coatings. As is seen, the values are quite respectable for magnet applications.

Table 1. Dielectric constant for sol-gel MgO-ZrO₂ insulation coating.

Thickness of Insulation (m)	Capacitance (F)	Capacitance with epoxy (F)	ϵ_{r1}	ϵ_{r2}
8.0x10-5	14 x10-11	17 x10-11	13	15
8.2x10-5	17 x10-11	22 x10-11	16	21
8.5x10-5	22 x10-11	27 x10-11	21	26
12x10-5	24 x10-11	34 x10-11	33	46

ϵ_{r1} :Dielectric constant without epoxy,

ϵ_{r2} :Dielectric constant with epoxy

Table 2. High voltage breakdown value for thin insulation coating.

Insulation coatings	Breakdown Voltage(kV)	Breakdown Current(mA)
ZrO ₂	0.6	20
YSZ	1	22
CeO ₂	1.2	19
MgO	1.2	11
SrTiO ₃	1	10
LaAlO ₃	1.1	12
PbTiO ₃	0.8	8
BaZrO ₃	1	14

Dielectric constant and high voltage breakdown values for the high temperature insulation coatings prepared by a continuous reel-to-reel sol-gel process have been measured by using a capacitive technique and Hubbel model 750-5 High Voltage Breakdown-AC Dielectric tester, respectively. The dielectric constant is about 20 without epoxy and about 25 with epoxy. These values compare reasonably well with the values reported in the literature for this material. High voltage breakdown values for the pinhole-free and crack-free thin insulation coatings is about 1 kV and 10 mA, which is respectable.

¹ Mutlu, I.H.; Celik, E. and Hascicek, Y.S, USA patent application pending, May, 1998.

High Strength Pure Copper Fabricated by Cryogenic Deformation

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The development of conductors with suitable combinations of strength and electrical conductivity is essential for the

fabrication of coils for high field magnets. A new cryogenic drawing process has been found to reduce the amount of dynamic recovery during fabrication of high strength pure copper conductors. The results of the processing technique have been reported in References 1 and 2. A major result of the research is the production of pure copper wire with a 295 K strength of 580 MPa and conductivity of more than 96% IACS. This strength level is about 45% higher than was previously available for pure copper. The material has a strength level of 680 MPa at 77 K and a resistivity ratio (resistivity at 295 K over that at 77 K) greater than six.

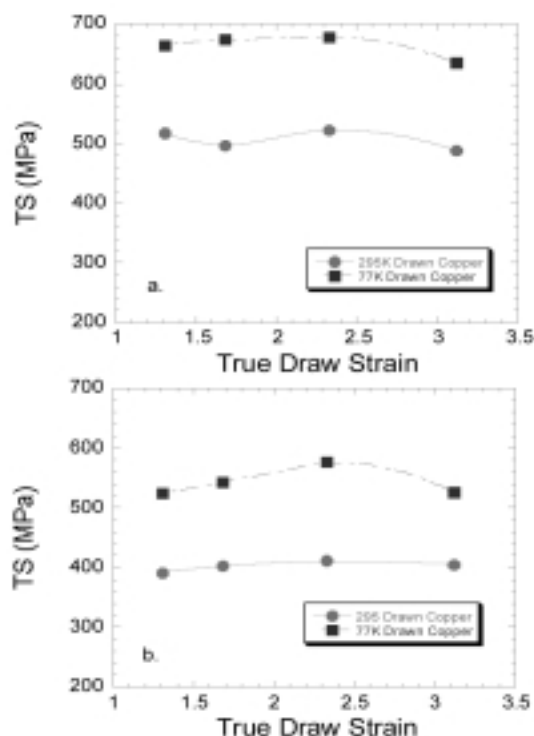


Figure 1. Strength (TS) variation with the drawing strains (ϵ) at (a) 77 K and (b) 295 K.

Figures 1A and 1B show tensile strength vs. drawing true strain (ϵ) for copper processed at 295 K and 77 K (where $\epsilon = \ln(A_0/A_f)$, A_0 and A_f are the initial and final cross-section areas). The maximum 295 K and 77 K tensile strengths are achieved for the materials drawn to a strain of 2.3 at 77 K. A positive work hardening rate is observed for 77 K deformed copper until true drawing strain reaches 2.3. Additional cryogenic deformation to a strain of 3.1 causes a decrease in both the 295 K and 77 K strengths. The strength decrease for high deformation strains is accompanied by a decrease in resistivity.

The decrease in strength is attributed to die geometry recovery effects, which may be related to room temperature annealing. Current studies focus on understanding and preventing or postponing of the softening phenomena. The optimization of the fabrication procedure has been undertaken and a further improvement of the properties of the cryogenically deformed pure copper has been achieved. The thermal instability of the cryogenically deformed Cu is addressed in post 77 K deformation, resistivity and annealing experiments. The 295 K

tensile strength of the cryogenically deformed copper (strain = 2.3) has been found to decrease after annealing at 100°C for 4 hours.

- ¹ Brandao, L., *et al.*, "New Cryogenic Processing for the Development of High Strength Copper Wire for Magnet Applications," to be published in *Adv. in Cryogenic Engineering, Materials*, 1999.
- ² Brandao, L., *et al.*, "Development of High Strength Pure Copper Wires by Cryogenic Deformation for Magnet Applications," to be published in *IEEE Transactions in Magnet Technology*, 1999.

Low Temperature Tensile and Fracture Toughness Properties of Niobium and Titanium

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A materials test program has been performed to support the structural design and analysis of the Accelerator Production of Tritium (APT) cavity assembly being designed at LANL. The materials studied are a high purity niobium (NbRRR250), commercially pure niobium (NbRRR40), commercially pure titanium (Ti Grade 2), and welds of the three metals. Materials having body-centered-cubic (BCC) crystal structure such as niobium are known to undergo ductile to brittle transitions making them undesirable for cryogenic applications. Titanium, has a hexagonal-close-packed (HCP) crystal structure and also has brittle tendency at low temperatures. Tensile properties can give indications of brittle behavior but do not provide fracture toughness design data. Tensile tests at 295, 77, and 4 K and fracture toughness tests at 4 K were performed to characterize the mechanical properties of the materials as a function of temperature. The fracture toughness test results (some of which represent first time measurements) are used to develop allowable flaw size criteria related to design and fabrication issues.

The 4 K mechanical properties test results are summarized in Table 1. The niobium tensile properties are shown as a function of temperature in Figure 1. The yield and tensile strengths increase dramatically upon cooling to 4 K from 295 K. The NbRRR250 retains ductility at 4 K, while the NbRRR40 is

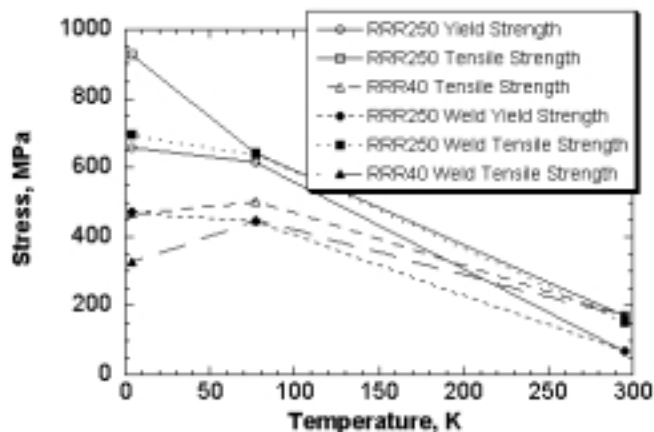


Figure 1. Niobium tensile properties vs. temperature.

extremely brittle exhibiting little plastic deformation in 4 K tensile tests. The decrease in tensile properties of the NbRRR40 at 4 K are attributed to embrittlement, which is in turn related to the higher impurity content of the NbRRR40 compared to the NbRRR250.

The NbRRR250 base metal has a 4 K toughness of 55 Mpa*m^{0.5} and the NbRRR40 material is 33 Mpa*m^{0.5}. The average 4 K toughness of the NbRRR250 weld and NbRRR40 weld are 26 Mpa*m^{0.5} and 32 Mpa*m^{0.5} respectively. The average 4 K fracture toughness of the titanium base metal and weld metal are 75 Mpa*m^{0.5} and 90 Mpa*m^{0.5} respectively. The tougher titanium weld metal was unexpected, and may not be accurate due to testing problems caused by residual stress in the welded plate.

The comparison of two grades of niobium shows there is a large effect of the purity on the low temperature properties. The lower purity niobium (NbRRR40) exhibits extreme brittleness in 4 K tensile tests, failing at a stress about one-half that of the NbRRR250. The most conservative design data with respect to the fracture toughness of niobium at 4 K is the minimum value (23 Mpa*m^{0.5}) observed for the NbRRR250 welded condition. The minimum 4 K fracture toughness data attained for the titanium is 64 Mpa*m^{0.5}, which is observed for the base material.

- ¹ Walsh, R.P., *et al.*, "Low Temperature Tensile and Fracture Toughness Properties of SCRF Cavity Structural Materials," proceedings 9th Annual Workshop on RF Superconductivity, 1999.

Table 1. 4 K mechanical properties test results.

Material	Yield Strength Mpa	Tensile Strength Mpa	Fracture Toughness* Mpa*m ^{0.5}
Nb (RRR250)	654 to 660	924 to 938	55 (+/- 6)
Nb (RRR250) Weld	466 to 473	592 to 779	26 (+/- 3)
Nb (RRR40)	N/A	433 to 525	33 (+/- 7)
Nb (RRR40) Weld	N/A	311 to 341	32 (+/- 3)
Ti Grade 2	926 to 945	1132 to 1165	75 (+/- 11)
Ti Grade 2 Weld	830 to 843	1116 to 1130	90 (+/- 2)**

* Toughness values are average of a minimum of 3 tests.
** Fracture toughness average of 2 tests.